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# Determination of the Attenuation Factor ($\beta$) in Hybrid Covalent/Non-Covalent Molecular Wires

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**Abstract:** We have established for the first time the molecular wire behaviour in a new set of hybrid covalent/supramolecular porphyrin-fullerene structures, in which hydrogen-bond interactions and *p*-phenylene oligomers of different length act as highly efficient molecular wires exhibiting a remarkably low attenuation factor ($\beta$ = 0.07 ± 0.01 Å$^{-1}$).

The design of suitably functionalized molecular wires is essential for developing the so-called *molecular electronics*.[1] A molecular wire is best described as a bridge that enables moving charges rapidly and efficiently over many chemical bond lengths. It guarantees an efficient electronic coupling between the electroactive termini. It implies that the bridge/wire exhibits an efficient matching between the donor and acceptor energy levels to realize charge transfer processes in the form of charge separation and charge recombination. Thus, it is important to probe the molecular wire behavior and to determine the efficiency of short- and long-range electron transfer processes. One of the key parameter of a molecular bridge is the attenuation factor ($\beta$), which determines the magnitude of the electronic coupling between redox sites as well as the energy of the electron (or hole) transfer states localized at the two termini.[2][3]

Two different approaches are known to realize efficient wire-like behaviour in electron donor-bridge-acceptor architectures. Firstly, covalent linkages in the form of π–conjugated bridges are used to couple electron donors and acceptors.[4] Representative examples are *p*-phenylenebutadiynylene (oPPB), oligo(*p*-phenylenevinylene) (oPPV), [2,2´] *p*-cyclophane-oligophenylenevinylene (*p*Cp-oPPv), oligo(*p*-phenyleneethynylene) (oPPE), oligofluorene (oFl), oligothiophene and polyporphyrins, to name a few.[5][6] In some cases, the molecular wire-like behavior has been assessed by determining $\beta$ in experiments. The values differ markedly between the different π-conjugated bridges and range from 0.25 (oPPB), 0.01 (oPPV), 0.012 (*p*Cp-oPPV), 0.2 (oPPE), 0.09 (oFl) to 0.003 (oTP) A$^{-1}$.[4][5] In general, lower $\beta$ values correlate with better molecular wire behavior. This allows the systematic analysis of a wide variety of molecular systems. Secondly, non-covalent means have been explored to realize electron donor-acceptor assemblies using, for example, electrostatic and/or hydrogen bonding interactions. By far the most compelling, non-covalent interactions are based on hydrogen bonds to modulate the electronic couplings between the redox sites. One of the first examples, in which hydrogen bonds have been demonstrated to control electron transfer in bischromophoric systems of zinc (II) and iron (III) porphyrins was reported by Therien *et al*.[7] Also, Hirsch *et al*. have reported on the use of a Hamilton-receptor/cyanuric acid motif to ensemble metalloporphyrins with $C_{60}$ derivatives.[8]

The wire behaviour of hybrid assemblies, in which the combination of a covalent π-conjugated bridge, on one hand, and non-covalent hydrogen bonds, on the other hand, constitute the binding between the redox sites has not been addressed to this date. Such a new approach to hybrid wires is a tremendous challenge and is likely to provide important incentives for the design of future molecular electronics.

Non-covalent, $C_{60}$-based hybrid ensembles, where the electron transfer processes occur exclusively through hydrogen-bonds in a *p*-(2-fulleropyrrolidinyl)benzoate and amidinium-ZnP complex (**1a•2b**) was reported by our research groups.[9] Inspired by this finding, we present herein a set of non-covalently associated $C_{60}$-*p*-phenylene oligomers, where the π-conjugation length of the oligomers in the bridges has been systematically altered. Particularly important in the design is the coupling of $C_{60}$ to the oligomeric bridge, in which the presence of a carboxylate as a terminus allows self-assembling with a porphyrin featuring an amidinium moiety through hydrogen bonds (Scheme 1). With such a molecular design at hand effects of distances and rates at which electron-transfer processes occur, as well as the molecular-wire behaviour based on π-conjugated systems and hydrogen bonds, were determined in our current study. It is important to note that this bio-inspired approach combining electron transfer through a supramolecular and covalent connectivity resembles charge transport in a variety of natural processes such as, for instance, photosynthesis[10] and cellular respiration.[11]

From the synthetic perspective, the asymmetric *p*-phenylene oligomers with an aldehyde and carboxylic termini are the key intermediates en-route towards pyrrolidino[3,4:1,2][60]fullerenes (**1a**-**d**). These oligomers were synthesized by Suzuki cross coupling reactions between bromoaryl and organoborane compounds in the presence of palladium (II) as catalysts and fluorine salts as base and additive, which is crucial for the success

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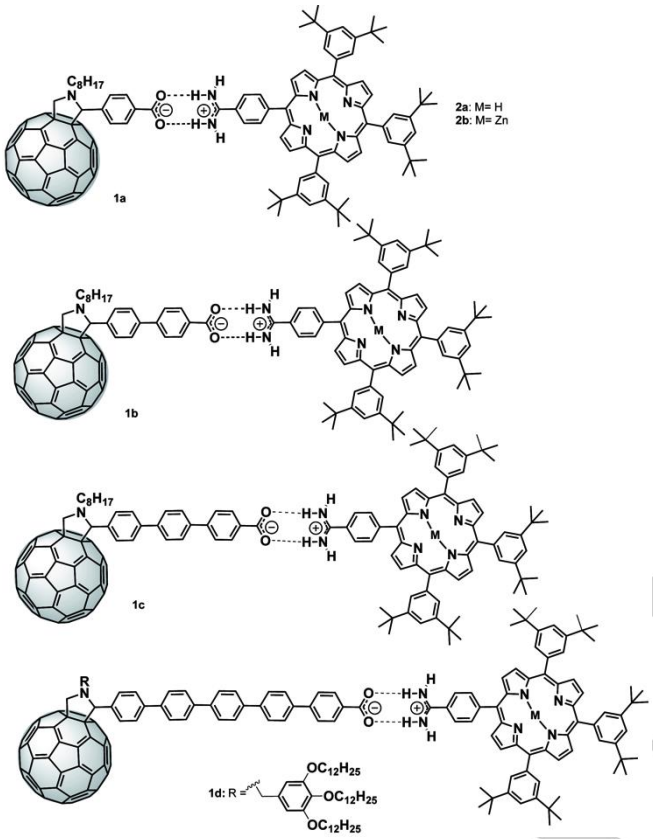
of the cross-coupling reaction.[12] Subsequent 1,3-dipolar cycloaddition reactions were employed to link the *p*-phenylene oligomers to $C_{60}$ through a pyrrolidine ring yielding **1a**-**d**, in high yields – for more synthetic details see supporting information.[13] On the other hand, **2a-b** were synthesized by using the conditions previously described by Ito *et al*.[14]




**Scheme 1.** Amidinium-carboxylate based ZnP•$C_{60}$ and $H_2$P•$C_{60}$ assemblies **(2a-b•1a-d)**.

Initially, we have employed absorption assays to evaluate the assembly of the non-covalently bound hybrids **1a-d**:**2a-b**. Figure 1 shows, as a representative example, the spectral changes of **2b** (2 x $10^{-6}$ M) upon addition of $C_{60}$ derivative **1a** (0-1.6 x $10^{-5}$ M) in toluene at room temperature. Depletion of the ZnP Soret- and Q-bands at 431, 530, 565, and 604 nm as well as bathochromically evolving transitions give rise to the appearance of eight isosbestic points. In the case of **2b** and **1a**, the emergence of a new transition at 442 nm is assigned to the formation of the complex **2b•1a**.

In addition, in the case of **2b•1a** new absorptions evolve in a range between 710 and 850 nm, where neither **2b** nor **1a** give rise to any appreciable features. Given the fact that **2b•1b**, **2b•1c**, and **2b•1d** lack the aforementioned absorption we postulate in line with recent investigations[15] the presence of a charge transfer state. The close spatial separation between **2b** and **1a** supports the notion of a shift of charge density from the electron donor to the electron acceptor (Figure S1).

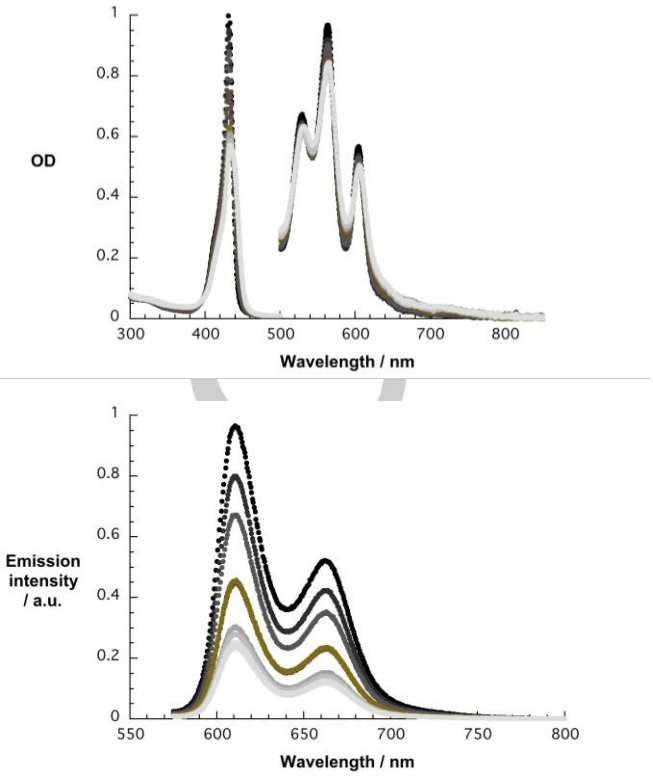



**Figure 1.** Upper part – Absorption changes observed during the complexation of **2b** (2 x $10^{-6}$ M) with **1a** (0-1.6 x $10^{-5}$ M) in toluene at room temperature. The 500-700 nm region is magnified by a factor of 20. Lower part – Normalized and fully corrected fluorescence spectra of **2b** (2 x $10^{-6}$ M) upon complexation with **1a** (0-1.6 x $10^{-5}$ M), photoexcited at 568 nm in toluene at room temperature.

As a complement to the aforementioned, we turned to steady-state fluorescence measurements of **2b** in toluene and chlorobenzene. To ensure full disaggregation of **2b** all experiments were carried out in the presence of 4-dimethylaminopyridine (DMAP) in a 1:1 ratio. To this end, excitation at either 436 or 567 nm, which match the absorption of the ZnP Soret- and Q-bands, respectively, provided insights into excited-state interactions in the presence of **1a**, **1b**, and **1c** (Figures S2-S4).

In particular, the intensity of the ZnP-centered fluorescence (Figure 1), with maxima at 615 and 665 nm and quantum yields of 0.04, was found to depend exponentially on the concentrations of added **1a**, **1b**, and **1c**. From the latter we infer the gradual transformation of **2b** into **2b•1a**, **2b•1b**, and **2b•1c**. Binding constants, which were obtained by nonlinear-least-square curve fitting of the ZnP fluorescence quenching integrated from 610 to 665 nm, are (2.4 ± 0.2) x $10^6$, (1.4 ± 0.3) x $10^6$, and (1.2 ± 0.3) x $10^5$ $M^{-1}$ for **2b•1a**, **2b•1b**, and **2b•1c**, respectively. Please note that these values are in excellent agreement with those derived from the absorption assays – *vide supra*. Interestingly the fluorescence quenching depends strongly on the bridge length, leading to a quenching factor of 71%, 24%, and 20% for **2b•1a**, **2b•1b**, **2b•1c**, respectively (Figures S2-S5).

Turning to the near-infrared part of the spectrum, we note for **2b•1a** a rather broad emission between 750 and 1200 nm (Figure S6). A deconvolution of this feature in chlorobenzene reveals the ZnP-centered fluorescence and phosphorescence at around 780 and 900 nm, respectively, as well as a charge transfer emission at 1047 nm. Excitation spectra of the 800 and 1047 nm give rise to maxima at 431 and 440 nm corresponding to **2b** and **2b•1a**, respectively. The charge transfer emission is subject to a solvent induced red-shift from 1003 nm in toluene to 1052 nm in THF, and broadening, that is, 185 nm in toluene to 235 nm in chlorobenzene. In stark contrast, the charge transfer emission is absent in **2b•1b**, **2b•1c**, and **2b•1d**.

Next, the corresponding ZnP•$C_{60}$ electron donor-acceptor hybrids were probed in transient absorption measurements in chlorobenzene upon 430 nm photoexcitation of **2b**. We had chosen the excitation wavelength of 430 nm and the ratio of 1:1:1 / **2b**:DMAP:**1a-d** to ensure excitation exclusively of **2b**. Starting with the **2b** photoexcitation in the ZnP reference – Figure S7 – its singlet excited state features evolve and they comprise maxima at 465, 545, 590, 645, 705, 1300 nm and minima at 565, 610 nm. From multiwavelength global analyses we derived a fast $S_2$ to $S_1$ transition within 4 ps and a first singlet excited state lifetime of 1.6 ± 0.2 ns. This decay reflects the ZnP intersystem crossing and the accordingly formed triplet excited state displays maxima at 480 and 840 nm.

When turning to the different ZnP•$C_{60}$ electron donor-acceptor hybrids, the same ZnP singlet excited state features are discernable during the early times. Notable is that here the transient features are the same, but the corresponding lifetimes differ substantially. For example, in **2b•1a** the ZnP singlet excited state decays within 0.64 ± 0.02 ns – Figure 2 – leading to a less efficient population of the ZnP triplet excited state. In addition, transient species evolve in the visible and the near-infrared region of the spectrum. Its features are, however, different from those of the singlet and / or triplet excited state of ZnP. In the visible, maxima at 475, 595, 645, 695 nm and minima at 570, 615 nm resemble what is known for the one electron oxidized form of ZnP. In the near-infrared, a 1010 nm maximum is a clear attribute of the one electron reduced form of $C_{60}$.

Taking the aforementioned into concert, we reach the conclusion that exclusive photoexcitation of ZnP in **2b•1a** is followed by an intramolecular electron transfer to afford the ($ZnP^{•+}$)•($C_{60}^{•-}$) radical ion pair state. Considering, however, the substantial overlay between ZnP singlet excited state and ($ZnP^{•+}$)•($C_{60}^{•-}$) radical ion pair state absorptions in the visible range we employed the 1300 nm ZnP singlet excited state absorptions as well as the 1010 nm radical anion absorption feature of $C_{60}$ to determine the electron transfer dynamics. The charge separation rates were deduced as 7.9 x $10^{10}$, 6.4 x $10^{10}$, 3.7 x $10^{10}$ and 2.4 x $10^{10}$ $s^{-1}$ for **2b•1a-d**, respectively, while charge recombination rates were 1.3 x $10^{9}$, 1.0 x $10^{9}$, 8.7 x $10^{8}$ and 6.0 x $10^{8}$ $s^{-1}$. Importantly, data deconvolution with target analysis[16][17] corroborates the charge separation and charge recombination trends (Figures S8-S13).

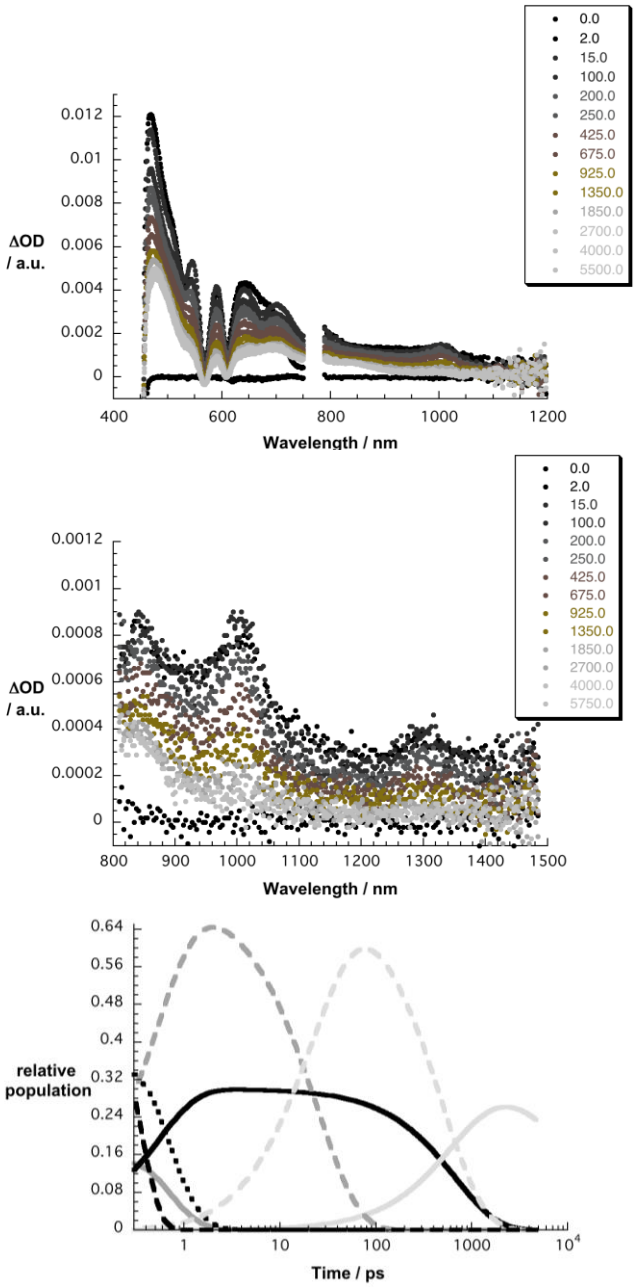



**Figure 2**. Upper part – Differential absorption spectra (visible and near-infrared) obtained upon femtosecond flash photolysis (430 nm) of **2b•1a** in chlorobenzene with several time delays between 0 and 5500 ps at room temperature. Central part – Differential absorption spectra (extended near-infrared) obtained upon femtosecond flash photolysis (430 nm) of **2b•1a** in chlorobenzene with several time delays between 0 and 5750 ps at room temperature. Lower part – Concentration time profile as obtained from target analysis showing ISC for unbound **2b** from the singlet excited state of **2b** (grey) to the triplet excited state of **2b** (dark grey) with a rate of 1.9 x $10^{-9}$ $s^{-1}$ and charge separation from the singlet excited state in **2b·1a** (red) to the radical ion pair state in **2b·1a** (purple) with a rate of 7.9 x $10^{10}$ $s^{-1}$ followed by charge recombination with a rate of 2.6 x $10^{9}$ $s^{-1}$.

With the electron rate constants in hand, we analyzed their dependence on the electron donor-acceptor separation, that is,

the distance between ZnP and $C_{60}$ in **2b•1a**, **2b•1b**, **2b•1c**, and **2b•1d**. From the corresponding slopes – Figures 3, S14, and S15 – $\beta$-values of 0.07 ± 0.01 $Å^{-1}$ were derived for the charge separation and charge recombination. Such low $\beta$-values, especially for *p*-phenylene oligomers, are due to the induction of co-planarization between adjacent phenylenes by the hydrogen-bonds.

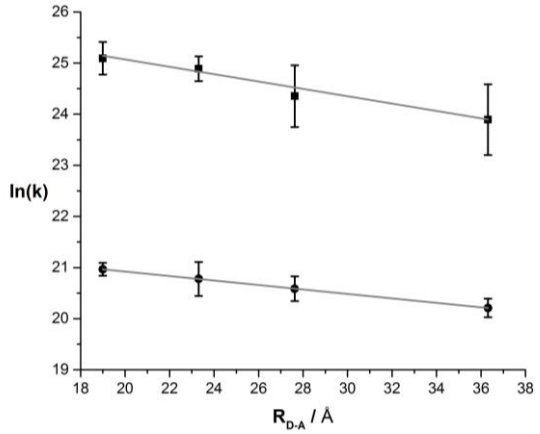



**Figure 3**. Dependence of charge separation and charge recombination on the center-to-edge distance in nitrogen-saturated chlorobenzene at room temperature for **2b•1a**, **2b•1b**, **2b•1c**, and **2b•1d**. The slope represents the $\beta$-values.

In summary, we have carried out the systematic synthesis of a new family of electron-donating ZnP and electron-accepting $C_{60}$ building blocks and their integration into electron donor-acceptor assemblies. For the first time, we have demonstrated the existence of an efficient charge transport through a combination of hydrogen-bonds and *p*-phenylene oligomers yielding an exceptionally small attenuation factor ($\beta$) of 0.07 ± 0.01 $Å^{-1}$. This is among the lowest $\beta$ values reported in the literature for molecular wires. As such, it validates the use of hybrid covalent/non-covalent wires for *molecular electronics* and, thus, paves the way to future designs employing a variety of known supramolecular interactions.

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**Keywords:** hydrogen bonding • molecular wire • electron transfer • fullerene

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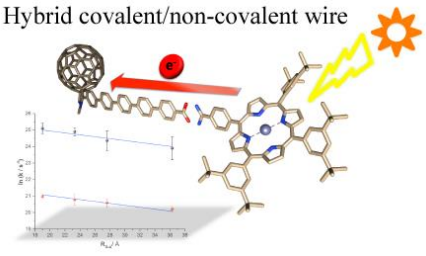
# COMMUNICATION

**Small Attenuation Factor in Hybrid Covalent and Supramolecular Wires**: A new series of electron donor-acceptor hybrids of ZnP and $C_{60}$ connected through covalent/non-covalent wires exhibit an efficient electron transfer process through hydrogen bonds and *p*-phenylene oligomers.

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**Determination of the Attenuation Factor (β) in Hybrid Covalent/Non-Covalent Molecular Wires**